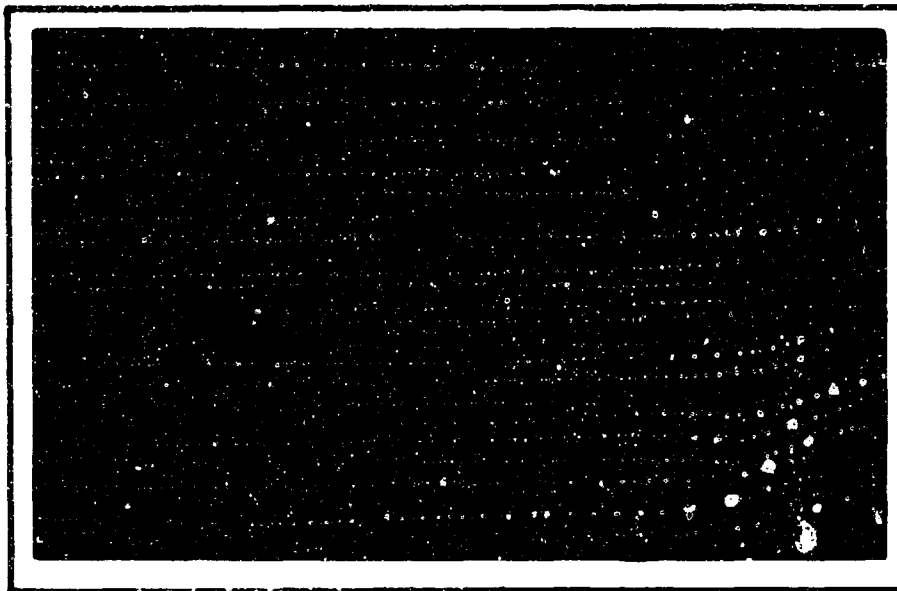


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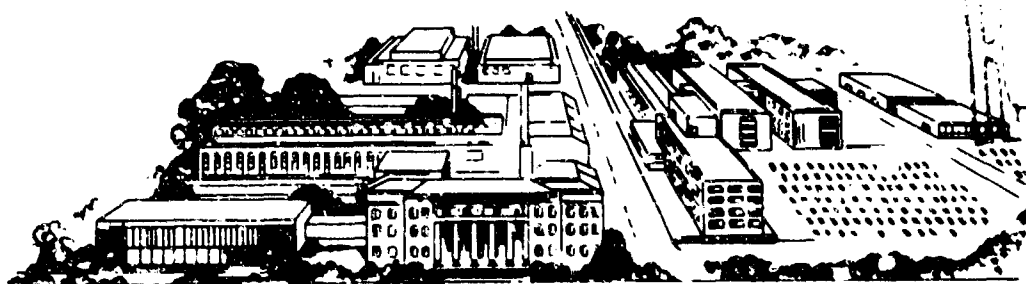
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FIRST QUARTERLY PROGRESS REPORT

on

STRUCTURAL CHANGES IN HIGH-STRENGTH  
STEEL ASSOCIATED WITH STRESS CORROSION  
AND ITS RELATIONSHIP TO DELAYED FAILURE

to

BUREAU OF NAVAL WEAPONS  
MATERIALS DIVISION - METALS BRANCH  
RRMA-223

September 28, 1964

by

D. A. Vaughan, D. I. Phalen, A. B. Tripler, and C. M. Schwartz

Contract No. N0w 64-0267-c  
Project No. WR-007-05-01

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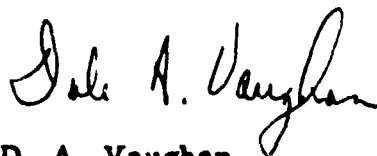
Gentlemen:

Contract No. NOw 64-0267-c  
Project No. WR-007-05-01

Enclosed is our first quarterly report, "Structural Changes in High-Strength Steel Associated With Stress Corrosion and Its Relationship to Delayed Failure", covering the period June 29 to September 28, 1964. Since final authorization to begin this program was delayed approximately 2 months after the contract date, the amount of experimental work conducted during the first quarter is limited. However, it is expected to proceed rapidly during the next quarter.

If there are any questions in regard to the experimental results, or the future work as planned, please contact me.

Very truly yours,



D. A. Vaughan  
Assistant Chief  
Physics of Solids Division

DAV:ng  
Enc.

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# STRUCTURAL CHANGES IN HIGH-STRENGTH STEEL ASSOCIATED WITH STRESS CORROSION AND ITS RELATIONSHIP TO DELAYED FAILURE

by

D. A. Vaughan, D. I. Fernald, A. B. Tripler, and C. M. Schwartz

## SUMMARY

The investigation of structural characteristics of AISI 4340 steel quenched and tempered to produce three strength levels has been initiated as a basis for the planned studies of these materials under conditions of stress-corrosion attack. Due to problems in contract negotiation, this program was delayed in starting. However, the experimental work, plus a literature study, has been initiated. Preliminary electron metallographic studies of the steel have been carried out in the process of developing techniques. Electron diffraction and X-ray diffraction results are being correlated with the microstructure.

## INTRODUCTION

Two months of the first quarter (June 29 to September 28, 1964) passed before negotiations were completed under this project, No. WR-007-05-01. However, some preliminary investigations are in progress which are directed toward establishing a base structure for AISI 4340 steel. X-ray and electron diffraction studies are being made to supplement electron and optical metallographic examinations of this steel which will be quenched and tempered to give three different tensile strength levels: 100, 180, and 260 ksi. These three conditions were selected to provide material of one composition but with variable susceptibility to delayed failure by stress-corrosion- or hydrogen-embrittlement-cracking mechanisms. Although no specific threshold strength level for susceptibility has been established in the steel, there is evidence that the susceptibility decreases with decreasing strength. It is important to note that the program is not designed to establish additional evidence of delayed damage, but instead is designed to identify the structural changes which are responsible for the degradation in the physical properties of this low-alloy high-strength steel.

The objective of the research to be conducted under this program is to investigate the effects of stress and corrosion (separately and combined) upon the crystal and grain structure of this important class of material (AISI 4340 steel) so as to elucidate the mechanism of stress-corrosion cracking. On the basis of an understanding of the fundamental processes which alter the structural properties of this steel, a fuller utilization of its inherent strength will be sought.

In addition to initiating the above experimental program, several published reviews on stress-corrosion cracking and on hydrogen-induced embrittlement in high-strength steel have been studied to assure a minimum of duplication and to become acquainted with the results of numerous evaluation programs on high-strength steels. A detailed discussion of the factors that influence the susceptibility of high-strength steel to hydrogen-induced brittle failures was presented by A. R. Elsea and E. E. Fletcher in a Defense Materials Information Center report No. 196. This report references over 100 publications on the relationships between hydrogen content and physical properties of the steel. It is concluded, therein, that all ferritic and martensitic steels are susceptible when tested under appropriate conditions; however, the high-strength steels are especially susceptible. Data are presented which indicate that failure occurs under a certain combination of applied stress, hydrogen content, and time. It is believed that the time factor is controlled by the movement of hydrogen through the steel and that the rate of hydrogen diffusion is influenced by the applied stress. Previous studies of the relationship between the brittle failure and microstructure have revealed that failure occurs only in body-center cubic material. However, details of the structural changes responsible for the failure have not been identified.

An understanding of the above mechanism is of considerable importance to the present program on stress-corrosion cracking of high-strength steel because of the potential of generating and introducing hydrogen into these steels by the corrosion process. An electrochemical analysis of the difference between stress-corrosion cracking and hydrogen embrittlement was presented by B. F. Brown's review of the problem in NRL Report No. 6041. He concludes that both of these processes occur on similar paths (prior austenite grain boundaries) but that this similarity in behavior cannot be used as a criterion to demonstrate that the same cracking mechanism is operative for the two phenomena. It is the objective of the research conducted under the current program to gain a fuller understanding of the mechanisms involved in these two cracking phenomena.

### STATUS OF EXPERIMENTAL WORK

For the initial investigation of structural-property changes associated with stress corrosion, specimens of AISI 4340 steel have been heat treated to develop three strength levels. Rolled sheet (0.065 inch thick) specimens were quenched from 1600 F and tempered at 400 F, 800 F, or 1300 F to give tensile strengths of approximately 260, 180, and 100 ksi, respectively. Additional specimens from the same sheet of 4340 steel, which had been tempered at 600 F, were available from another program. This last material was used to develop the preliminary background described in this report.

Mechanical and stress-corrosion fractures of this material were examined by optical and electron metallographic techniques. Electron microscopic examination by the replica technique appears to be more useful than direct optical examination. Figure 1 shows a typical example of the surface topography resulting from a mechanical fracture, while Figure 2 shows the typical block structure on a fractured surface resulting from stress corrosion. An interpretation of these structures is premature at this time, but they illustrate the marked difference in fracture path.



18,750X

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FIGURE 1. ELECTRON MICROGRAPH OF MECHANICALLY FRACTURED  
SURFACE OF AISI 4340 STEEL

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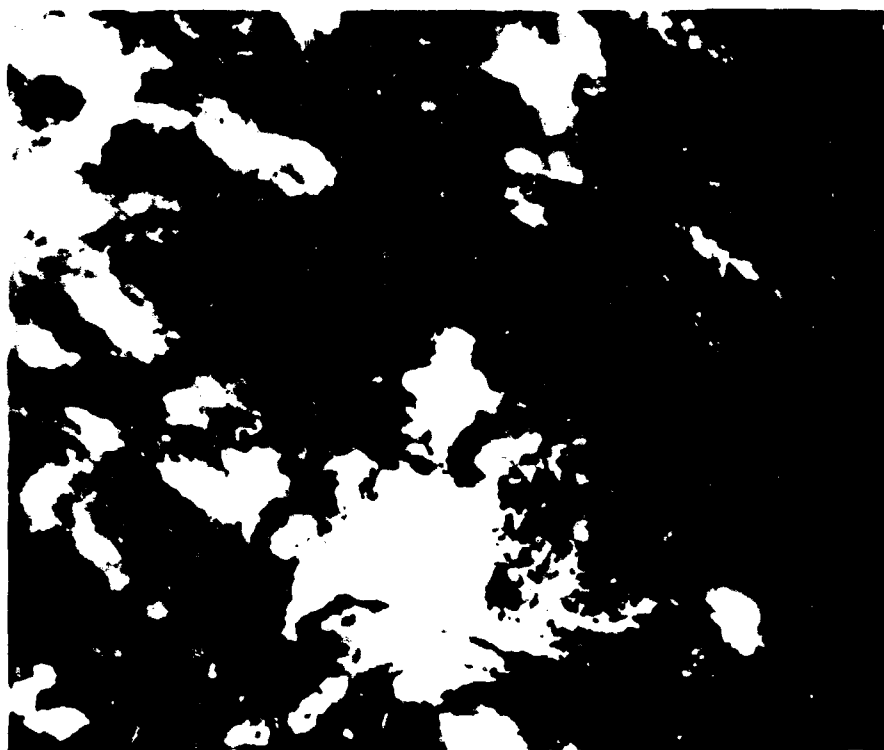
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FIGURE 2. ELECTRON MICROGRAPH OF STRESS-CORROSION FRACTURE  
IN AISI 4340 STEEL





46,000X

J11171

a.



107,000X

J11173

b.

FIGURE 3. TRANSMISSION ELECTRON MICROGRAPHS OF 4340 STEEL  
QUENCHED FROM 1575 F AND TEMPERED AT 600 F

Since the above structures reveal surface contours at the fracture rather than the internal structure, thin sections of the steel were prepared by electropolishing techniques. The initial examinations did not reveal structures that could be related to those observed in either Figure 1 or Figure 2. However, considerable difficulty was encountered in retaining a section of the metal thin enough for transmission electron microscopy. This has been attributed to atmospheric attack which destroyed the very thin portions of the metals so that large areas could not be examined. The internal structure revealed by transmission electron micrographs, Figures 3a and 3b, is exceedingly complex. Undoubtedly, part of this complexity is artifact resulting from the atmospheric attack. The grain size computed from the average diameter of the light areas in Figure 3b is approximately 1000 Å, while the boundaries appear to be of the order of 100 Å in width. However, there are large areas, see Figure 3a, in which scattering of electrons within the grains results in a network of fine structure. This structure may be due to tangles of dislocation, strain contours, carbide precipitates, artifact, and/or retained austenite. Electron-diffraction examination of these thin sections revealed the large-grain phase to be beta martensite, which gives a spot pattern of body-centered cubic symmetry. The pattern contained additional diffraction spots, some of which could be identified as belonging to austenite, while others are believed to be due to a carbide phase. X-ray diffraction examination did not reveal the austenite phase. The diffraction lines of the beta martensite were broadened by micro-strain and/or crystallite size, both of which may be expected in quenched and low-temperature-tempered 4340 steel. A more complete analysis of this type in quenched and tempered specimens of 4340 steel will be made as this program progresses.

#### FUTURE WORK

The investigation of the base structure will be continued on the AISI 4340 heat treated to three strength levels. It is planned to explore methods for protecting the thinned section of steel from atmospheric attack so as to limit unwanted corrosion reactions. Subsequent studies would then be made after intentional anodic and cathodic corrosion reactions. X-ray and electron diffraction examinations will be extended to obtain more fundamental data on crystal structure of these materials.

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